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13. ABSTRACT (Maximum 200 words)

Hall and Seebeck measurement systems have been specified, purchased, facilitated and calibrated. These systems are assisting in research efforts to develop new analytical approaches, based on electronic tools which have served the electronic industry well, to assess structural materials used in defense assemblies and structures. These electronic systems will develop multiple measurements techniques to assess alloy phase stability, aging , microstructure and properties. Seebeck coefficient measurements have already been correlated with the diffusible hydrogen content in alloy with high transition metal element contents. These results show promise for assessment of hydrogen effects in gun barrel steel, naval nickel aluminum bronze fasteners, and high strength steel welds. Hall and Seebeck measurements have been used to characterize the microstructure in alloy thin films. Research is in progress to develop electronic techniques to determine the susceptibility of sigma formation in superalloys. These electronic techniques will offer rapid non destructive microstructural assessment with quantitative data acquisition for easy long term storage of product quality assurance.

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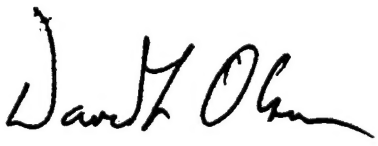

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FINAL REPORT

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Introduction

The use of electronic and magnetic analytical instrumentation to assess structure-property relationships as they relate to needs of the electronic industry has made it possible to produce high quality and reproducible devices. The ability to make property determination and quality control has made it possible. There have been significant advancements in these electronic and magnetic instrumentation as seen by their measurement sensitivity, reduction in instrument size, electronic controls and data acquisition and most important a sound fundamental understanding the correlation of the measurement with electronic and phase structure. Opportunities are now arising for the use of these established analytical instruments and techniques for phase and microstructure determination, phase stability and prediction of service life of structural materials, such as found in high performance engines, vehicles and weapon systems. The advent of non destructive evaluation of microstructure and properties is offering significant savings in both money and time in maintaining many defense systems.

The project was to purchase the electronic analytical systems for use as both for electronic property characterization and for the development of practices to assess microstructure, phase determination, phase stability and the prediction of remaining service life of structural component. The instruments purchased, which will be used with the existing comprehensive optical and electronic microscopy, x-ray diffraction and surface analytical systems at the Colorado School of Mines, are listed below:

DURIP Equipment Summary

The equipment purchased is consistent with the proposed instruments described in the proposal.

1. Hall Measurement System

Manufacturer: Lake Shore Cryotronics, Inc., 64 E. Walnut St., Westerville, Ohio, 43081-2399; Fax: (614) 891-1392; Telephone: (614) 891-1392

1. 7507 Low Voltage Autoswitching, High Sensitivity Hall measurement system with sample holder and cards for samples from 1 ohm to 150 gigaohm and in a

temperature range from 77 K to 400 K. High output magnet power supply and Neslab CFT-300 recirculating chiller

2. 75014 Closed Cycle Refrigerator Sample Module with a compressor, rated for a temperature range between 15 K and 350K.
   Total cost of system: $179,953.

2. Seebeck Measurement System

Manufacturer: MMR Technologies, Inc. (1400 North Shoreline Boulevard, Suite A-5, Mountain View, CA 94043-1346; Fax: (650) 9629647; Telephone: (650) 962-9620

1. Seebeck Effect Measurement System, the model K20SB100-5R includes K-20 Programmable Temperature Controller with control software, two Extended Wide Temperature Range Thermal Stage Units, SB100 Seebeck Electronics and software (no computer)
2. Hardware for vacuum and pressure manifolds and control, computer interface (from assorted vendors), required for custom pumping arrangement.
   Total cost of system: $22,440

Both systems, which are permanently installed at CSM in Meyer Hall, have been tested, calibrated and are fully functional. The initial measurements on a wide variety of samples have shown promising results. For instance measurements on TiB_x - TiC and $CrSi_x$ thin films show low Hall mobility but very high carrier concentration ($\sim 10^{23}$ cm^{-3}) for samples of low resistivity. In addition measurements on highly resistive, undoped CdTe and CdS thin films as well, have been made to demonstrate the full range of the system.

Our ability to measure conductive metal films with the Hall measurement system has also been performed and has demonstrated that it can produce high quality results that are very useful for our current and proposed studies in the development of advanced alloys based on electronic based alloy theories. The measurement of the hydrogen content in materials using the Seebeck measurement system is becoming an every day practice. Projects for which this equipment will be used are listed below. The project numbers indicate investigations that are currently funded by DoD.

1. PHACOMP Meter - Sigma Phase in Turbine Engines

New PHACOMP, the electron-based calculation technique, has been used to investigate nickel-based weldments. These calculations are important for predicting the tendency of austenitic alloys to precipitate topologically close-packed phases which adversely affect mechanical and corrosion properties. Since Phacomp and New Phacomp computations are based on the electronic filling of the d-band, Seebeck coefficient measurements should offer a direct measurement practice to determine the tendency of having a phase change in the these alloys. This new

electronic characterization capability allows for the development of direct non-destructive evaluation of the phase stability of parts in service, such as turbine blades. (ARO DAAD 19-01-1-0375)

2. **Advanced Hydrogen Sensor for Materials**
   Both diffusible and trapped hydrogen contents are being measured in hydrogen storage materials and structural alloys. Seebeck measurements have been found to offer a rapid assessment of the hydrogen contents in alloys with substantial transition element contents. Techniques are been developed for steel weldments and gun barrel steel under ARO contract and an initial effort is under way to assess the hydrogen content in aluminum bronze fasteners, which were hydrogen charged by cathodic protection, for the U. S. Navy.
   (Steel weldments; ARO DAAD 19-00-1-0149, barrel steel for Benet Arsenal, Marine fastners: U. S. NSWC).

3. **Characterizing Thin Film Microstructure**
   The new equipment is being used to characterize thin film microstructure to develop predictive models for evolution of thin film microstructure and properties. These measurements will form an integral part of the graduate education for physics, metallurgy and materials science, as well as supporting research activities in the CSM Advanced Coating and Surface Engineering Laboratory.

4. **Colloboration with Los Alamos National Laboratory**
   New NDE techniques for evaluating the metallurgical aging of plutonium alloys. The current project makes plutonium measurements at LANL, but surrogate materials have been measured with the Seebeck measurement system at CSM to assist in designing the LANL equipment..

5. **Collaboration with U.S. Naval Surface Warfare Center NDE R and D Group**
   The equipment will be used in collaboration with Robert Denale (Head, Welding and NDE R&D U.S. Naval Surface Warfare Center) to measure diffusible hydrogen associated with welding of high strength steels. The high strength steel weld metal program is being performed by other investigators under ONR Grant N00014-94-1-0694.

6. **Collaboration with Army Laboratory**
We have had discussions with U.S. Army Benet Laboratory in the use of advanced methods to measure hydrogen content in steels in gun barrels. Our new instrumentation will allow preliminary NDE studies with the guidance of Paul Cote (Benet Laboratory).

7. **Measurement of properties of transparent conducting oxide coatings**
Transparent conducting oxide coatings form an important part of display technologies, photovoltaics and window coatings. Rapid electrical characterization with Hall and Seebeck will be used to characterize and develop materials with superior properties.

8. **Studies of semiconductor thin films for photovoltaic applications**
The processing stages of photovoltaic modules have great impact on solar cell efficiency and stability. The cell properties will be correlated with measurements on both cells and sub-modules consisting of layers of the actual devices. This work is supported by DOE through its Thin Film Partnership.

ACKNOWLEDGEMENTS:

The numerous investigators, who will be frequently using these instruments to further their work, will acknowledge the support of the DOD Equipment grant program.